

Robert P. Yunick
Vice President
Corporate Technology

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August 18, 1997

Certified Mail
Return Receipt Requested
Z 231 069 781

Chemical Inventory Section
OPPTS 7407
Room G-099
U.S. EPA
401 M Street, SW
Washington, DC 20460



22970000548

Attn: Chemical Inventory

RECEIVED
08/21/97
97 AUG 22 PM 3:10

Dear Sir or Madam,

Based on the suggestion of Mr. Kent Anapolle of EPA, Schenectady International, Inc. (SII) presents this third-party filing of a TSCA inventory correction for a compound known as para-dodecylphenol (PDDP).

To our knowledge, based on Chemical Abstracts Service's (CAS) electronic database, there are 32 listed CAS Registry Numbers for dodecylphenol. Two of these numbers are or have been in recent use to represent PDDP currently distributed in commerce. They are:

27193-86-8	Phenol, dodecyl-
104-43-8	Phenol, 4-dodecyl-

Both of these CAS RNs are listed in the U.S. TSCA Inventory, Canadian DSL and European EINECS list.

The 27193-86-8 RN represents a straight-chain C_{12} substituent without specifying its position on the phenol ring. The 104-43-8 RN represents a dodecyl phenol with a straight-chain C_{12} substituent in the para position. Unfortunately, neither of these RNs or any of the other 30 known RNs accurately represent the structure of PDDP currently in commerce, nor in commerce in 1979 when the TSCA Inventory was created.

Commercial PDDP is a complex mixture of primarily para isomers of highly branched C_{12} substituents. It is made by alkylating phenol with an olefin known as dodecene which is made by oligomerization of propylene (propylene tetramer). Given the branched structure of the C_{12} substituent in commercial PDDP, it does not match the straight-chain structure in either of the Registry Numbers.

Some recent filings of PMNs by SII customers using SII's PDDP as a reactant has focused attention on this issue, and led to discussions with Mr. Anapolle. His cooperation in understanding the past history and complexity of CAS RN assignments, and allowing this third-party filing to aid in the PMN processing is much appreciated.

In 1986, SII was confronted with a similar situation with para-nonylphenol (PNP) which is made using propylene trimer. SII was part of an industrial group negotiating a voluntary TSCA testing consent order with EPA. The issue of identifying the test substance brought forth the realization that the Registry Numbers in use at the time for PNP were for straight-chain substituents, and not representative of the branched-chain commercial product. SII filed an inventory correction for branched PNP. The issues related currently to PDDP are very similar to those that were confronted in 1986 for PNP.

~~TSCA CONFIDENTIAL~~
~~BUSINESS INFORMATION~~
~~DOES NOT CONTAIN NATIONAL~~

NOT CBI.

2/28/19

SII (then known as Schenectady Chemicals, Inc.) began manufacture of PDDP in 1976, but only on an intermittent basis. It was not until 1989 that continuous production was commenced. Since 1976 and continuing to date, PDDP manufacture has occurred at SII's Plant No. 2 at our facility in Freeport, TX. The same propylene tetramer source and essentially the same process has been used through the years. Minor changes in process have been incorporated to improve process efficiency or product quality.

Attached is a copy of our 1976 Technical Data Sheet (S-1143 (2/76)) showing the properties of the one grade of PDDP manufactured at that time; and a more recent version (S-1143, R5, 7/11/95) currently in use which shows properties for two grades: a Lube Grade and a Standard Purity Grade.

Also attached is a copy of a gas chromatogram (2/29/96) of PDDP showing the complex mixture of isomer peaks. This chromatogram was used as part of a presentation I made on March 7, 1996 to the Interagency Testing Committee (ITC) at the request of Dr. John D. Walker, Executive Director of the ITC. This presentation was made to the ITC following the ITC's listing of PDDP among several alkylphenols in the ITC's 37th Report. The presentation covered the composition and manufacture of commercially available alkylphenols.

Since 1989, SII has used high-performance liquid chromatography (HPLC) to monitor PDDP composition for quality control purposes. HPLC is more expeditious than the gas chromatography method.

In order to fulfill the request of Mr. Anapolle that we demonstrate that the current commercial product is comparable to that made previously, I submit the following:

- 1) The 1976 Technical Data Sheet shows properties very similar to those in the current version (last revised 1995).
- 2) Based on past analysis of competitors' PDDP, it is our belief that all commercially available PDDP is made from propylene tetramer. The PDDP may differ slightly in content of ortho-isomer, para-isomer and didodecylphenol, but otherwise are very similar. These variations are believed caused by small variations in alkylation and purification processes from manufacturer to manufacturer.
- 3) Due to intense price competition on the sale of PDDP, and a review of available olefins and their cost, we conclude that propylene tetramer is the only cost-competitive olefin that can be used to make PDDP; and further that every PDDP manufacturer in the 1970's used propylene tetramer as the olefin of choice, as is done today.
- 4) We have no data or reason to conclude that PDDP manufactured today differs substantially from PDDP manufactured in the 1970's. Small variations exist depending on how a particular refinery makes propylene tetramer; and how each PDDP manufacturer operates its alkylation and purification processes, but to our cumulative knowledge today's product involves the same raw materials and essentially the same process as when the TSCA Inventory was created.

More specifically to SII's own case, I submit the following HPLC data to show the composition of product manufactured in 1989 and 1997. Enclosed are the following:

- 1) Analytical Method HO41168 - Assay of p-Dodecylphenol by HPLC - of May 1989.
- 2) Fax of May 8, 1989 from Blair Janes to R. Gillespie conveying the method to our Texas manufacturing site. Mr. Janes characterized the method as provisional and still under development.
- 3) Copy of two HPLC strip charts for July 24, 1989 showing the analysis of a production lot (left chart, Run #597) and a chromatograph standard (right chart, Run #619).
- 4) A May 15, 1990 revision (now obsolete) of the HO41168 Analytical Method.
- 5) A current (October 14, 1996) version of the HO41168 Analytical Method.
- 6) Certificate of Analysis for batch no. T7-3684 of PDDP of August 4, 1997.
- 7a) HPLC strip chart (Run #259) for PDDP standard of August 4, 1997.
- b) HPLC strip chart (Run #260) for the first drum of batch no. T7-3684 of August 4, 1997.
- c) HPLC strip chart (Run #261) for the last drum of batch no. T7-3684 of August 4, 1997.

To help you understand these chromatographic results, let me offer the following. It is customary practice for an analyst to use a so-called "standard" when using HPLC as an assay method. While the 1989 version of HO41168 does not describe the making or using of a standard, the 1990 and 1996 revisions do include it.

A standard is a contrived mixture of known analytes that are likely to be found in the sample undergoing analysis. In 1989, the PDDP standard consisted of the ortho- and para-isomers of dodecylphenol and phenol. When the method was revised in 1990, the standard had in it those two isomers as well as phenol and didodecylphenol. More recently, phenyl ethers have been added.

Therefore, if you examine the 1997 standard (Run #259), the top peak which was not integrated is an artifact related to system solvent, and the following peaks listed by elution time are:

<u>Elution Time, min.</u>	<u>Component</u>
2.43	Phenyl ethers
2.95	Didodecylphenol
4.11	ortho-Dodecylphenol (two peaks)
5.40	PDDP
8.90	Phenol

Based on that standard, the 1997 analyses and 1989 analysis assayed as follows (numbers expressed as percent):

<u>1997 First Drum Run #260</u>	<u>1997 Last Drum Run #261</u>	<u>1989 Sample Run #597</u>	<u>Component</u>
--	--	--	Phenyl ethers
--	--	0.667	Didodecylphenol
5.409	5.414	2.753	ortho-Dodecylphenol
94.591	94.586	96.580	PDDP
--	--	--	Phenol

Thus, between 1989 and 1997, the makeup of SII's PDDP has changed slightly in the following ways: 1) didodecylphenol is no longer present in measurable quantity; 2) the para content has declined as a result by about two percent; and 3) ortho content has increased accordingly.

Let me also point out that when you compare the 1989 chromatograms (product assay and standard) to 1997 chromatograms (products and standard) the elution times for respective components differ, i.e., the 1989 PDDP peak eluted at about seven minutes, while in the 1997 chromatograms it eluted at 5.4 minutes.

The reason is attributed to the fact that the 1989 method was considered provisional and still under development. It used an ethanol/heptane solvent system. When it was revised in 1990, due to further method refinement, the elution times changed and are in closer agreement with those indicated in the 1996 method, and in the actual 1997 data. The major change was the use of an iso-octane/isopropanol/acetic acid solvent system. The column support material was also changed.

Despite what the 1996 method lists for expected elution times, slight variations still occur. These may be caused by variances in solvent ratio, operator technique, as well as other variables. Because of these variables, we run a standard for comparison to assure the identity of peaks in the chromatogram.

Based on the above information and the enclosures, SII asks that EPA correct the TSCA Inventory to reflect the branched nature of PDDP. We ask also that this be done in a way to qualify the corrected name/structure for listing in the Canadian DSL and European EINECS list. We ask also that customers' PMNs that are currently in suspense due to this issue be taken off suspense and resume processing.

Given the manner in which the para-nonylphenol correction was made for CAS RN 84852-15-3*, an appropriate, suggested Inventory name for commercial PDDP could be:

Phenol, 4-dodecyl-, branched

If you have any questions, or need further information, please contact Mrs. Dorothy M. Kraus at 518-347-4401 or by fax at 518-382-8129 or by e-mail at dmksii@transit.NYSER.NET; or me at 518-347-4411, same fax number, or by e-mail at robert.yunick@siigroup.com. Mrs. Kraus participated in the PNP Inventory correction and is familiar with this PDDP request.

Thank you for your cooperation in processing this request.

Very truly yours,

SCHENECTADY INTERNATIONAL, INC.



Robert P. Yunick
Vice President
Corporate Technology

Attachments

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RECEIVED

DODECYLPHENOLTYPICAL PHYSICAL PROPERTIES

Appearance	Clear, pale yellow viscous liquid.
Molecular Weight (calc.)	262
Boiling Point @ 760 mm	
°F	633
°C	313
Viscosity, cps	
@ 25°C	3250
@ 50°C	288
Flash Point (Cleveland Open Cup)	
°F	310
°C	154
Hydroxyl Number	218

TYPICAL ANALYSIS

p.-Dodecylphenol	92%
o.-Dodecylphenol	6%
Didodecylphenol	2%

SPECIFICATIONS

Color (APHA)	200 max.
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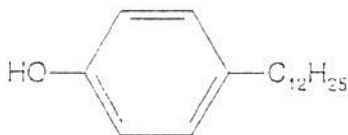
AVAILABILITY

Dodecylphenol is available in bulk in tankwagons or in 55-gallon (208 liter) drums, 425 lbs. (192.7 kg.) tankcars, or net weight.

S-1143 (2/76)

TECHNICAL DATA

PARA-DODECYLPHENOL



CAS RN	27193-86-8
Molecular Formula	C ₁₈ H ₃₀ O
Molecular Weight	262.4

TYPICAL PHYSICAL PROPERTIES	
Appearance	Clear, viscous liquid
Boiling Point (@760mm)	313°C (633°F)
Viscosity @25°C	2500-4000 cps
Hydroxyl Number	200-220
Specific Gravity @ 25°C	0.94
Flash Point (COC)	>160°C (>320°F)

SPECIFICATIONS		
	Lube Grade	Standard Purity
Dodecylphenol	90.0% min.	97.0% min.
Color (APHA)	500 max.	100 max.
Moisture Content ppm	1000 max.	500 max.

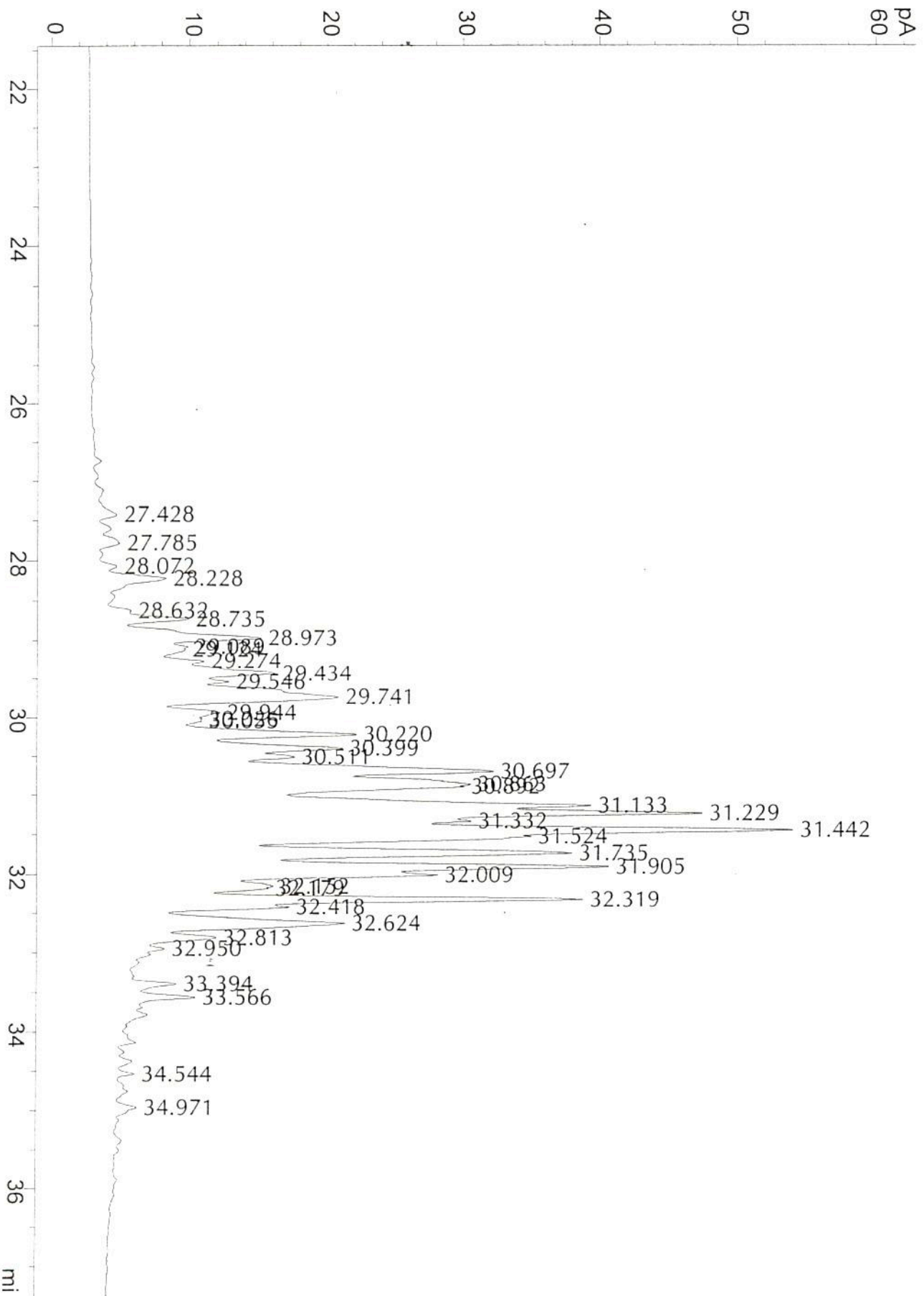
AVAILABILITY

Dodecylphenol is available in bulk in tankwagons or tankcars, and in 55 gallon drums (208 liter) at 425 lbs (192.7 kg) net weight.

S-1143 R5 (7/11/95)

Para - Dodecylphenol

FID1 A, of 2-29-96\011F0101.D



SCHENECTADY CHEMICALS INC.

P.O. Box 1046, Schenectady, N.Y. 12301
(518) 370-4200, Telex No. 145-457, Cable SCHENVAR

ANALYTICAL DEPARTMENT

METHOD: H041168

TITLE: ASSAY OF p-DODECYL PHENOL BY HPLC

APPARATUS:

INSTRUMENT: Isocratic HPLC with UV detector and electronic integrator

Column: Dupont Zorbax CN; 25 cm x 4.6 mm ID

ANALYSIS PARAMETERS:

Mobile Phase: 0.7% ethanol (absolute) in Heptane (HPLC grade)
Use a 1 L volumetric flask to prepare mobile phase. Pipet 7 mL of ethanol into flask and fill to the mark with Heptane. Mix and vacuum filter through a 0.45 micron filter. Do not all mixed solvent to stand under vacuum (NOTE 1).

Flow Rate: 2.0 mL/min.
Injection Volume: 20 uL
UV wavelength: 280 nm

SAMPLE PREPARATION: Accurately weigh approximately 50 mg of sample into a 25 mL volumetric. Dissolve and dilute to the mark with Heptane.

METHOD OF CALCULATION: Area Normalization with response factors
Response factors are derived from the multi-component standard.

ORDER OF ELUTION (approximate):

Didodecyl phenol= 4 to 5 min.
o-dodecyl phenol= 5 to 6.5 min.
p-dodecyl phenol= 8 to 11 min.

NOTES:

1. The retention times are very sensitive to mobile phase composition. The purpose of vacuum filtering is two-fold. It filters any particulate matter which can clog the system and it removes dissolved gases from the solution. HPLC grade solvents are now generally pre-filtered and if care is taken when preparing the mixture the filtration step can be omitted. However a de-gassing step is recommended. This can be accomplished by sparging helium through the solution for one minute. The helium sparge should not be left on, however because it will cause the solvent composition to change.

SCHENECTADY CHEMICALS, INC.

W. Howard Wright Research Center
2750 Balltown Road
Schenectady, New York 12309 USA

TELEFAX LEAD SHEET

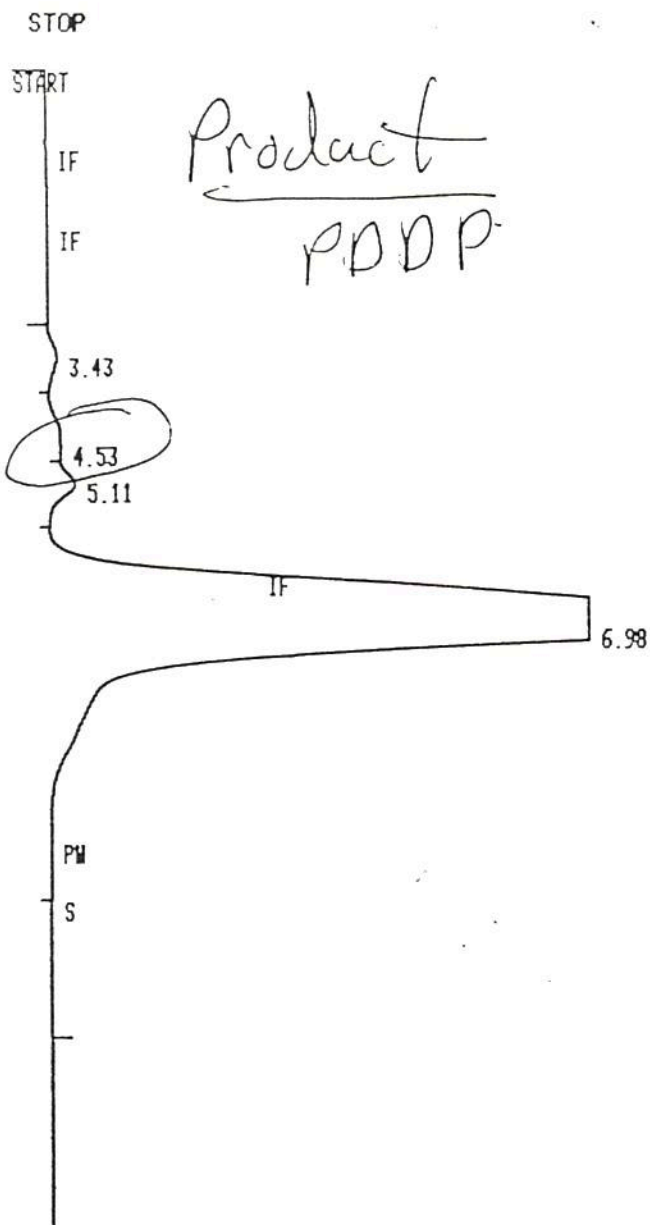
Date May 8, 1989 From Blair Janes
To R. Gillespie
Location SCI - Texas Phone 518-370-4200
Fax Number Fax 518-382-8129

SPECIAL INSTRUCTIONS/MESSAGE

1. "Attached" is provisional HPLC method for assay of P-dodecylphenol. Work on finalizing method is in progress and should be done soon.
2. Regarding your offer to arrange accommodations, please make lodging reservations for Paul Deltry. Arrival: Sunday 5/14 (guarantee room for late arrival) Leaving: Wednesday, 5/17.

Thanks,

Blair



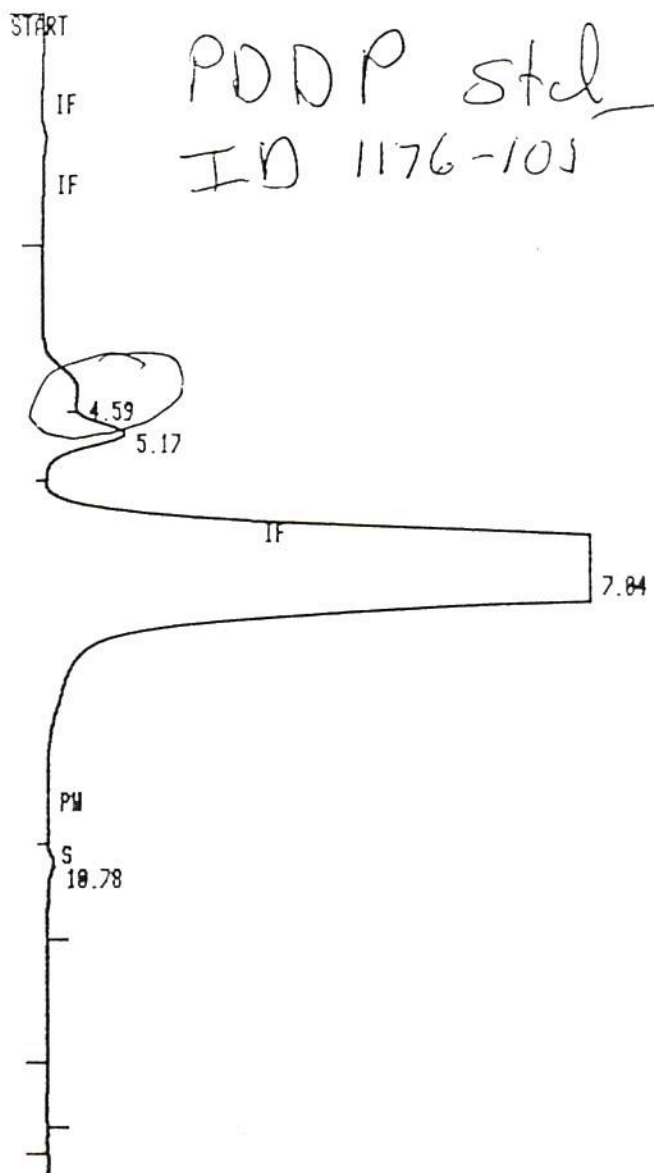
RUN # 597
WORKFILE ID: B1
WORKFILE NAME:
SAMPLE #100

JUL/24/89 02:16:59

AREA%	RT	AREA TYPE	AR/HT	AREA%
	3.43	135920 BY	0.507	0.662
→	4.53	219220 VV	0.578	1.076
	5.11	341450 D VH	0.433	1.677
	6.98	1.9670E+07 SHB	0.838	96.580

TOTAL AREA= 2.8367E+07
MUL FACTOR= 1.0000E+00

VIAL # 100
INJECTION# 1 OF 1



RUN # 619
WORKFILE ID: B1
WORKFILE NAME:
SAMPLE #100

JUL/24/89 12:57:37

AREA%	RT	AREA TYPE	AR/HT	AREA%
	4.59	496790 PY	0.513	1.517
	5.17	1062000 D VH	0.457	3.243
	7.04	3.1114E+07 SHB	0.812	95.018
	10.78	72559 BB	0.355	0.222

TOTAL AREA= 3.2745E+07
MUL FACTOR= 1.0000E+00

VIAL # 100
INJECTION# 1 OF 1

M*: 17HP 1898L SCI=TX METHOD

SCHENECTADY CHEMICALS INC.

P.O. Box 1046, Schenectady, N.Y. 12301
(518) 370-4200, Telex No. 145-457, Cable SCHENVAR

OBSOLETE

ANALYTICAL DEPARTMENT

Test No.	H041168
Date Issued	June 16, 1989
Date Revised	May 15, 1990
Reason for Revision	Change of chromatography conditions

Assay of Para-Dodecylphenol by HPLC

APPARATUS

Instrument: Isocratic HPLC with UV detector and electronic integrator

Column: Whatman Partisil 10 PAC; 25 cm x 4.6 mm ID (Part no. 4225-001)

ANALYSIS PARAMETERS

Mobile Phase: 98/2: 2,2,4-trimethylpentane/2-propanol, 1% glacial acetic acid.

Measure 98 parts by volume 2,2,4-trimethylpentane (iso-octane) and 2 parts by volume 2-propanol (isopropanol). Mix the two solvents. Add 1.0 mL of glacial acetic acid to each liter of 98/2 solvent. Mix and vacuum filter through a 0.45 micron filter. Do not allow solvent to stand under vacuum (NOTE 1).

Flow Rate:	2.0 mL/min.
Injection Volume:	20 uL
UV Wavelength:	280 nm

Elution Times (approximate):	
Didodecylphenol=	3 to 4 min.
o-Dodecylphenol=	4 to 5 min.
p-Dodecylphenol=	5 to 6 min.
Phenol=	8.75 min.

METHOD OF CALCULATION

Area Normalization with response factors. Response factors are derived from a multi-component standard.

SAMPLE PREPARATION

Accurately weigh approximately 50 mg of sample into a 25 mL volumetric. Dissolve and dilute to the mark with HPLC grade 2,2,4-trimethylpentane.

STANDARD PREPARATION:

Using materials of known purity prepare a multi-component standard at the following concentrations:

Phenol=	0.012 mg/mL
Didodecylphenol=	0.016 mg/mL
o-Dodecylphenol=	0.012 mg/mL
p-Dodecylphenol=	1.92 mg/mL

CALCULATION OF RESPONSE FACTORS

1. Inject the multi-component standard three times or until agreement of the integrated areas is within 3% relative.

2. Calculate the response factor of p-Dodecylphenol(PDDP):

$$K = \frac{(\text{PDDP average area}) 100}{(\text{conc. PDDP, mg/mL})(\% \text{ PDDP purity})}$$

3. Calculate normalized response factors for remaining components.

$$K_n = \frac{(\text{component conc., mg/mL})(\% \text{ component purity})(K)}{(\text{average component area}) 100}$$

The normalized response factor for PDDP is one, by definition

4. The response factors should be checked every two hours of continuous operation. The normalized response factors should be updated when a difference of 5% from the previously calculated factors occurs.

CALCULATION OF ACTUAL SAMPLE CONCENTRATION

Inject sample solutions at least twice or until the agreement of the integrated peak areas is within 3% relative. Multiply the integrated areas of each component by their respective normalized response factors, K_n . The individual component percent is then calculated from these corrected areas:

$$\% \text{ component} = \frac{(\text{average integrated area})(K_n) 100}{\text{sum of corrected areas}}$$

Test No.

H041168

OBSOLETE

NOTES:

1. The retention times are very sensitive to mobile phase composition. The purpose of vacuum filtering is two-fold. It filters any particulate matter which can clog the system and it removes dissolved gases from the solution. HPLC grade solvents are now generally pre-filtered and if care is taken when preparing the mixture the filtration step can be omitted. However a de-gassing step is recommended. This can be accomplished by sparging helium through the solution for one minute. The helium sparge should not be left on, however, because it will cause the solvent composition to change.

SAFETY: Material Safety Data Sheets (MSDS) are available for reagents used and materials analyzed. The analyst is responsible for taking all safety precautions. No one is to perform any test if he/she is unsure of proper safety precautions. If the analyst has any concerns, he/she shall discuss these concerns with supervision before proceeding.

The statements and information given in this analytical procedure are for general use only by qualified individuals and should not be relied on when establishing appropriate procedures for the recipient's own operation. The accuracy or completeness of all statements, technical information and recommendations contained herein are not guaranteed and no warranty of any kind is made in respect thereto. In addition, this literature contains information which Schenectady Chemicals, Inc., considers proprietary. The original recipient of this information from Schenectady Chemicals, Inc., is not authorized to further disseminate it without written permission from Schenectady Chemicals, Inc.

Author: Todd K. Hulse 5/15/90
Approved By: A. Brian Jones 6/1/90
Manager, Analytical Department

6/90

ASSAY OF *p*-DODECYLPHENOL BY HPLC

1.0 APPLICATION AND DESCRIPTION

This procedure is designed to determine the purity of *p*-dodecylphenol. High pressure liquid chromatography is used to determine the percentage of *p*-dodecylphenol, *o*-dodecylphenol, didodecylphenol, and phenyl ethers. Response factors are determined for each component. The response factors are applied to the normalized area percent data.

2.0 REFERENCES

None

3.0 INTERFERENCES

There are no known interferences with this analysis.

4.0 EQUIPMENT/APPARATUS

In all cases other brands or models of equipment may be substituted if deemed equivalent in specifications to those given.

- Liquid Chromatograph: Hewlett-Packard 1090L with 280 nm detector capability.
- Chromatography Column: Whatman Partisil 10 PAC; 25cm x 4.6 mm ID (Part No. 4225-001).
- Electronic analytical Balance: 0.0001 g capable; Mettler, Model AK 160
- Integrator: Hewlett Packard Integration System Model #3390 or higher, or Hewlett Packard Chem Station
- Chromatography vials: 2 mL; Hewlett-Packard, part no. 5180-4197
- Volumetric Flasks: 25 mL Class A; Pyrex brand, Fisher Scientific part no. 10-211A
- Pasteur pipettes: 9 in., disposable; Kimble part no. 72050

5.0 REAGENTS

- 2,2,4-Trimethylpentane (isooctane): EM Science Omnisolv® PX1834, SII Item No. 901009
- 2-Propanol (iso-propyl alcohol): EM Science Omnisolv® PX1834, SII Item No. 6004
- Glacial acetic acid: A.C.S. reagent grade, Fischer Scientific A-38, SII Item No. 1167
- *p*-Dodecylphenol; SII Item No. 400155
- *o*-Dodecylphenol; SII Item No. 4000018
- Didodecylphenol; SII Item no. 400157
- Phenol; SII Item No. 4021

6.0 SAFETY

The analyst must wear the personal protective equipment (PPE) required for handling samples of PDDP.

Material Safety Data Sheets (MSDS) are available for reagents used and materials analyzed. The analyst is responsible for taking all safety precautions and reviewing all new chemicals' MSDS prior to working with them. No one is to perform any test if they are unsure of proper safety precautions. If the analyst has any concerns, they shall discuss these concerns with their supervision.

7.0 INSTRUMENT/EQUIPMENT SET UP PROCEDURES

7.1 Chromatograph Operating Parameters

Mobil Phase: 2,2,4-Trimethylpentane/2-propanol (0.1% glacial acetic acid); 98/2 v/v

Measure 98 parts by volume of 2,2,4-trimethylpentane and 2 parts by volume 2-propanol. Mix the two solvents. Add 1.0 mL glacial acetic acid to each liter of 98/2 solvent. Mix and vacuum filter through a 0.45 μ filter. Do not allow the solvent to stand under vacuum¹.

Flow Rate: 2.0 mL/min.
Column Oven Temperature: ambient
Injection Volume: 20 μ L
Detector: 280 nm

Approximate Elution Times:

Ethers	1.5 min.
Di-dodecylphenol (DDDP)	2.9 min.
<i>o</i> -Dodecylphenol (ODDP)	4.1 min.
<i>p</i> -Dodecylphenol (PDDP)	6.1 min.
Phenol	9.6 min.

7.2 Integrator Parameters

Run Time: 13 min.

- C. Approximate Elution Times (minutes)
An example chromatogram is shown in Figure 1.

¹ The retention times are very sensitive to mobile phase composition. The purpose of vacuum filtering is two fold. It filters any particulate matter which can clog the system and it removes dissolved gases from the solution. HPLC grade solvents are now, generally, pre-filtered and if care is taken when preparing the mixture the filtration step can be omitted. However, a de-gassing step is recommended. This can be accomplished by sparging helium through the solution for one minute. The helium should not be left on, however, because it will cause the solvent composition to change.

8.0 INSTRUMENT CALIBRATION

The liquid chromatograph is maintained per the manufacturers instructions. No calibration of the instrument is indicated.

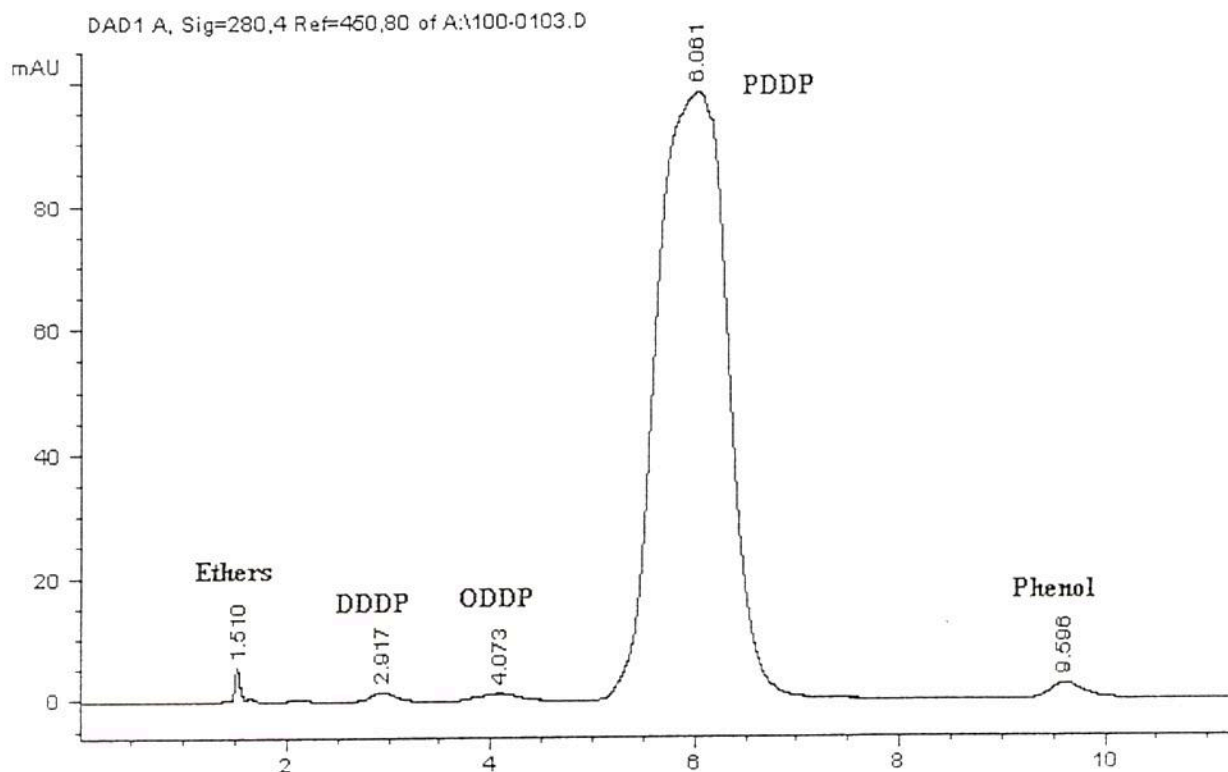


FIGURE 1

9.0 STANDARD PREPARATION

- 9.1 Prepare a standard by weighing the following component amounts into a 100 mL class A volumetric flask: Dilute to volume with 2,2,4-trimethylpentane
- Phenyl ethers: 0.02 g
 - Di-dodecylphenol: 0.016 g
 - Ortho-dodecylphenol: 0.012 g
 - Para-dodecylphenol: 1.92 g
 - Phenol: 0.012 g
- 9.2 The standard mixture is used to determine a response factor for each component of the standard. Once response factors are established they may be used indefinitely. The response factor calibration should be checked by injecting the standard solution once and calculating response factors. If they agree within 5% of the previous calibration the response factors are still valid.

10.0 ANALYSIS PROCEDURE

10.1 Sample preparation

Prepare the sample at a concentration of 2.0 mg/mL. For example: weigh 50 mg of sample into a 25 mL volumetric flask. Dissolve and dilute to the mark with 2,2,4-trimethylpentane.

10.2 Inject the multi-component standard three times or until integrated area agreement of 3% is achieved. The integrated areas from one injection must agree within 5% of the previous calibration.

10.3 Inject the sample twice or until integrated area agreement of 3% is achieved.

11.0 CALCULATIONS

11.1 Response Factor

Using the data from the multi-component standard, obtain the average integrated area for each component. Apply the following equation to obtain the response factor for *p*-dodecylphenol (PDDP):

$$K = \frac{(PDDP \text{ average area})(100)}{(PDDP, \text{ mg/mL})(\%PDDP \text{ purity})}$$

11.2 Calculate normalized response factors for remaining components.

$$K_n = \frac{(\text{component conc., mg/mL})(\% \text{ component purity})(K)}{(\text{average component area})(100)}$$

The normalized response for PDDP is 1.

11.3 Multiply the integrated areas of each component by their respective normalized response factors, K_n , to obtain the corrected area. The corrected area for each component is divided by the sum of the corrected areas for all components:

$$\% \text{ Component} = \frac{(\text{average integrated area})(K_n)(100)}{\sum \text{ of corrected areas}}$$

12.0 REPORTING RESULTS

Report all results to the number of significant figures required by the specifications.

13.0 PRECISION

	MDL	RDL	RSD (N=30)	Confidence Limits $\alpha=0.01$
ETHERS	0.15%	0.36%	14.4%	$\pm 0.030\%$
PHENOL	0.02%	0.06%	28.6%	$\pm 0.005\%$
PDDP	NA	NA	0.2%	$\pm 0.092\%$
ODDP	NA	NA	3.9%	$\pm 0.116\%$
DDDP	0.09%	0.22%	8.6%	$\pm 0.019\%$

MDL The method detection level (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

RDL The reliable detection level (RDL) is that level above which the risk of reporting a false negative is 1%. A false negative means reporting the absence of the analyte when it is actually present.

RSD The relative standard deviation (also known as coefficient of variation). The RSD is the mean divided by the standard deviation times 100.

Confidence Limits

The 99% confidence limits around an average within which the true value will lie. The given values were calculated from data where the analyte was at or near the specification limits.

14.0 REMARKS

None

15.0 DISCLAIMER

The statements and information given in this test procedure are for general use only by qualified individuals and should not be relied on when establishing appropriate procedures for the recipient's own operation. The accuracy or completeness of all statements, technical information and recommendations contained herein are not guaranteed and no warranty of any kind is made in respect thereto. In addition, this literature may contain information which Schenectady International, Inc., considers proprietary. The original recipient of this information from Schenectady International, Inc. is not authorized to further disseminate it without written permission from Schenectady International, Inc.

16.0 SIGNATURES

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Manager, Analytical Department

Manual Title: Work Instructions (Tier IV)
Section: Analytical Department Analysis Methods
Procedure: H041168

Page: 6 of 6
Revision: 1
Date Issued: October 14, 1996

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REVISION/REVIEW FORM

REVISION NUMBER	REVISION DATE	REVISION/REVIEW EXPLANATION & DETAILS	APPROVAL
1	October 14, 1996	New procedure in controlled format	ABJ

1ST LAST AVG

QUALITY RESULTS

Moisture Content ppm

193.89	106.96	144.93
--------	--------	--------

A.P.H.A. Color

5.0	5.0	5.0
-----	-----	-----

Assay (wt. percent) by LC

Phenol

— — — — —

Ortho-Dodecyl phenol

5.409	5.414	5.415
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Para-Dodecyl phenol:

94.591 94.586 94.5885

Di-Dodecyl Phenol

(,	,
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Miscellaneous

—	—	—
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Retain has been taken? YES

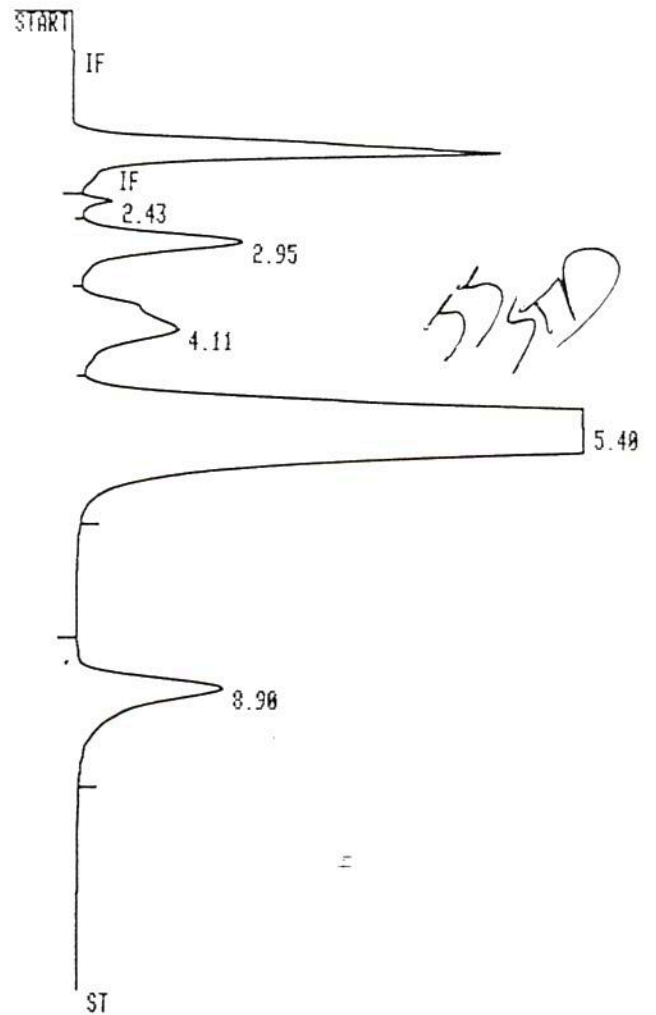
Railcar or Tankwagon No: DRUMS

Analyst: CW

Date: 08.04.97

Note: Run LC standard and attach.

WKFILE 1 @



RUN # 259
WORKFILE ID: A1
WORKFILE NAME:
SAMPLE #100

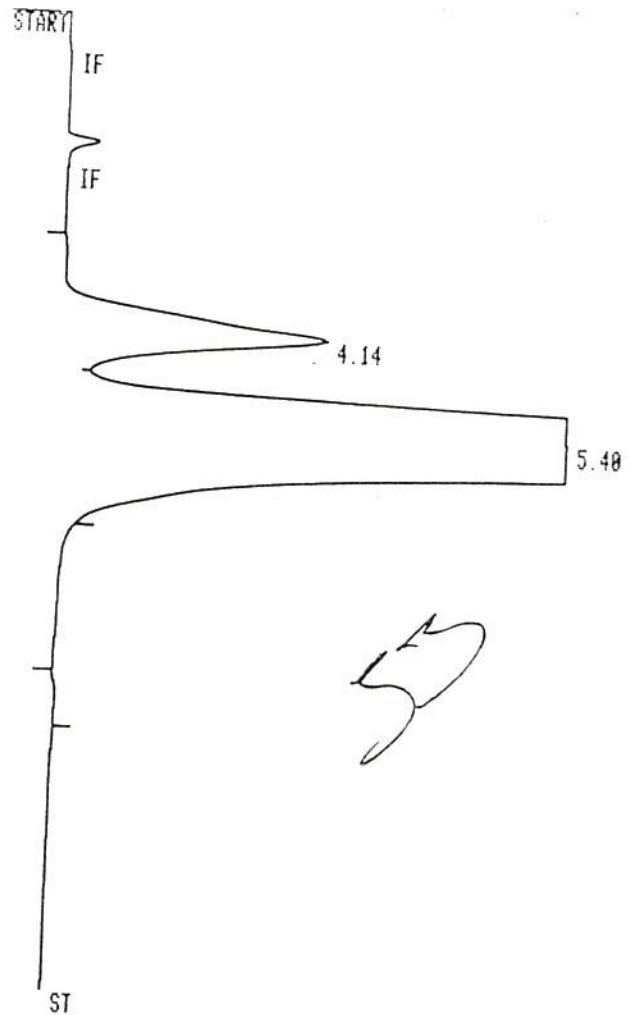
AUG/04/97 12:12:15

NORM

RT	AREA	TYPE	CAL #	AMOUNT
2.43	46513	D PY		0.003
2.95	636190	VP	1R	4.336
4.11	706150	PV	2R	4.851
5.40	1.2952E+07	V8	3R	87.336
8.90	931360	FB	4R	3.475

DELETE WKFILE 2
STORE WKFILE 1 2

1st ↓
Drum



RUN # 268
WORKFILE ID: A1
WORKFILE NAME:
SAMPLE #100

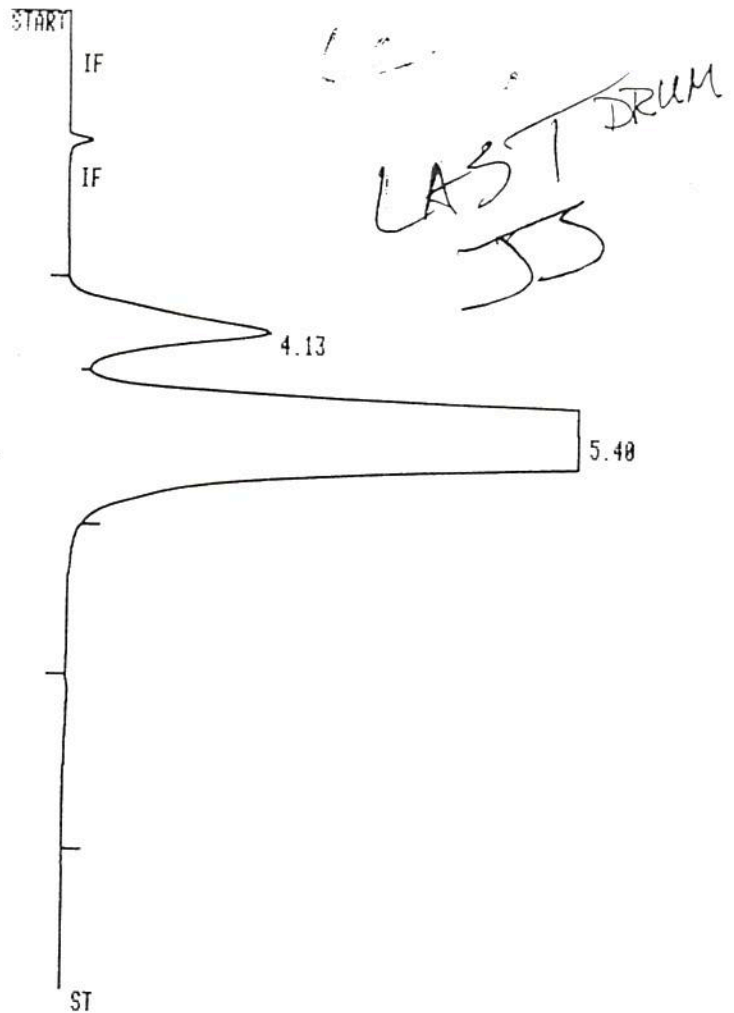
AUG/04/97 12:33:35

NORM	RT	AREA	TYPE	CAL #	AMOUNT
	4.14	1709700	PV	2R	5.489
	5.48	3.0461E+07	VB	3R	94.591

TOTAL AREA= 3.2171E+07
MUL FACTOR= 1.0000E+00

VIAL# 100
INJECTION# 1 OF 1

M# : PDDP LG SII=TEX METHOD



RUN # 261
 WORKFILE ID: A1
 WORKFILE NAME:
 SAMPLE #100

AUG/04/97 12:50:41

NORM

RT	AREA	TYPE	CAL #	AMOUNT
4.13	1307500	PV	2R	5.414
5.40	2.3271E+07	VB	3R	94.586

TOTAL AREA= 2.4579E+07
 MUL FACTOR= 1.0000E+00

VIAL# 100
 INJECTION# 1 OF 1

NOT CBI

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Smid
2/28/19